

## 2-(2-Chloroethoxy)-ethyl Acetate and 2-Chloroethyl Vinyl Ether

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Dioxane is the principal impurity in the crude 2-chloroethyl vinyl ether prepared by the method of Cretcher.<sup>2</sup> Cretcher considered that the two formed an azeotrope which boiled at 107°. This azeotrope appeared remarkable in that its boiling point was between those of the two components of the azeotropic mixture.

In the work reported here, chloroethyl vinyl ether was prepared in 60% yield by Cretcher's method. When the crude product was distilled through a column having 60 theoretical plates, dioxane was obtained at 101–102°, a mixture of dioxane and ether at 102–108°, and finally, pure ether at 108°. Since both pure dioxane and pure ether were distilled from the mixture, it is evident that no azeotrope was formed.

The ether was also distilled at reduced pressure (120 mm.). Dioxane distilled at 52–53°, and chloroethyl vinyl ether at 59°; a mixture of variable composition was obtained between the pure components.

The following properties were observed with chloroethyl vinyl ether<sup>3</sup>: b. p., 108°, 59° (120 mm.);  $n_{D}^{20}$  1.4378;  $d_4^{20}$  1.0475.

(2-Chloroethoxy)-ethyl Acetate.—An effort was made to produce chloroethyl vinyl ether by the pyrolysis of 2-(2-chloroethoxy)-ethyl acetate. Since the latter is a new compound, its preparation and pyrolysis are described. Diglycol chlorohydrin was acetylated with acetic anhydride, and the product (94% yield) was purified by distillation: b. p. 80° (1 mm.);  $d_4^{20}$  1.1546;  $n_{D}^{20}$  1.4398. Found:  $M^{20}_D$  38.02; C, 43.3; H, 6.8. Calcd.:  $M^{20}_D$  38.07; C, 43.3; H, 6.7. Pyrolysis over Pyrex glass at 500 and 550° (contact time, 8 sec.) decomposed 32 and 83%, respectively, of the ester but produced little if any chloroethyl vinyl ether. Most of the products were gases.

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(2) Cretcher, Koch and Pittenger, *THIS JOURNAL*, **47**, 1173 (1925).

(3) Cretcher reported b. p., 108° (740 mm.);  $d_4^{15}$  1.0525; W. Chalmers reported b. p. 108°,  $n_{D}^{20}$  1.4362;  $d_4^{20}$  1.044 (*Can. J. Research*, **7**, 464 (1932)).